

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 08-337493

(43)Date of publication of application : 24.12.1996

(51)Int.Cl.

C30B 15/10
// C30B 29/06
H01L 21/208

(21)Application number : 07-148737

(71)Applicant : SUMITOMO METAL IND LTD

(22)Date of filing : 15.06.1995

(72)Inventor : ARAHORI TADAHISA

(54) HIGH-PURITY GRAPHITE MEMBER FOR PULLING UP SINGLE CRYSTAL AND ITS PRODUCTION

(57)Abstract:

PURPOSE: To easily obtain a high-purity graphite member which is less contaminated by impurities and from which the production of a silicon single crystal having high quality is possible without requiring a special equipment by subjecting the graphite member to be purified to a heat treatment in a specific reduced pressure range and temp. region.

CONSTITUTION: The desired graphite member is obtd. by heat treating the graphite member to be purified in the temp. region of 1500 to 2500° C under the reduced pressure of 10⁻¹ to 10⁻⁶Torr. This graphite member is the high-purity graphite member for pulling up the single crystal having a metal impurity content of ≥1ppm. The reason why the metal impurity content is confined to 1ppm lines in that if this content exceeds 1app the purification of the graphite member is insufficient and the silicon single crystal having the required high quality (high purity) is not obtainable with the device for pulling up using the graphite products consisting of such member. The metal impurities, such as Fe, Ni, Cr, Cu and Na, in the graphite member which are considered harmful for semiconductors are evaporated by this treatment and are removed from a purification treating furnace by a vacuum pump, etc.

LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

* NOTICES *

JPO and NCIPi are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] The high-purity-graphite member for crystal pulling characterized by a metal impurity content being 1 ppm or less.

[Claim 2] The manufacture approach of the high-purity-graphite member for crystal pulling characterized by heat-treating purified material under reduced pressure of 10^{-1} - 10^{-6} Torr in a 1500-2500-degree C temperature region.

[Translation done.]

* NOTICES *

JPO and NCIPi are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the graphite member for crystal pulling and its manufacture approach of the high grade used when manufacturing the single crystal of semi-conducting material, such as silicon.

[0002]

[Description of the Prior Art] From the former, it faces producing semi-conducting material, especially a silicon single crystal, and the Czochralski method called a Czochralski process (it is hereafter described as a CZ process) is used widely.

[0003] Drawing 1 is drawing showing typically the general crystal manufacturing installation used by the CZ process. As shown in this drawing, the closed-end cylinder-like graphite crucible 1 is supported by the support shaft 2 which rotates at a predetermined rate, and the heater 3 and the heat insulating mould 9 are arranged in the outside of a graphite crucible 1 by concentric circular. Similarly inside a graphite crucible 1, the closed-end cylinder-like quartz crucible 4 is installed, and it fills up with the melting liquid 5 of the silicon raw material which heats and carried out melting at the heater 3 inside the quartz crucible 4. Furthermore, on the medial axis of the quartz crucible 4, the raising shafts 6, such as a wire which rotates at the rate of predetermined, are arranged in the support shaft 2, this direction, or hard flow with the same axial center as the support shaft 2. And the single crystal 8 with which melting liquid 5 congeals and is formed is grown up by contacting the seed crystal 7 attached at the head of the raising shaft 6 on the front face of melting liquid 5, and pulling up the raising shaft 6.

[0004] In recent years, with high integration of LSI, the request to quality improvement of silicon is strong, and much more high grade-ization is demanded also about the purity of a graphite member in connection with this. In case this manufactures a silicon single crystal by the CZ process, it is because the carbon material in a pull-up furnace is also exposed to an elevated temperature, and the impurity in a carbon material evaporates, it remains in a furnace and it has big effect on the quality (purity) of silicon.

[0005]

[Problem(s) to be Solved by the Invention] Generally the graphite material as a member for a silicon pull-up is manufactured through each process of shaping, baking, graphitization, and purification. Although the impurity mixed into graphite material while passing through these production processes was removed at the last purification process, as the approach of this purification, it was common after graphitization to have performed elevated-temperature halogen gassing.

[0006] However, a 5-20 ppm impurity is contained in the graphite material obtained by the above-mentioned conventional approach, and purity is inadequate as graphite material for high quality silicon manufacture.

[0007] In addition to a limitation being in the purification effectiveness, since this conventional approach used staining substances, such as strong acid and halogen content gas, for purification processing, it needed a special reactor and a special wastes treatment equipment, and it caused lowering of productivity, and a cost rise.

[0008] About the approach of raising the purity of graphite material, the crystal pulling equipment which used for each part the approach of heat-treating, for example, supplying halogen gas under

reduced pressure and the graphite material obtained by this approach is proposed (JP,63-79759,A, JP,6-2637,B, and JP,7-29762,B). Technical problems, such as the aforementioned cost rise, have come [however, / the approach indicated here has a facility and a complicated process, and] to be solved.

[0009] Since the conventional elevated-temperature halogen gassing of high-grade-izing is inadequate, the purification effectiveness is excellent, a metal impurity content can manufacture the graphite material of a high grade 1 ppm or less, and this invention is made considering offering an approach simple also in facility, and cheap as a technical problem.

[0010]

[Means for Solving the Problem] The summary of this invention is in the high-purity-graphite member of following (1), and its manufacture approach of (2).

[0011] (1) The high-purity-graphite member for crystal pulling characterized by a metal impurity content being 1 ppm or less.

[0012] (2) The manufacture approach of the high-purity-graphite member for crystal pulling characterized by heat-treating purified material under reduced pressure of 10^{-1} - 10^{-6} Torr in a 1500-2500-degree C temperature region.

[0013] In the pull-up equipment used by the CZ process indicated to be the aforementioned graphite member to drawing 1 , the graphite product used for a crucible, a heater, incubation heat insulation, etc. is said.

[0014] Moreover, a metal impurity content means Fe, nickel, Cr, Cu, Na, K, calcium, and the other sum total contents of all the metallic elements contained as an impurity.

[0015]

[Function] The metal impurity content of the high-purity-graphite member of this invention (invention of the above (1)) is a graphite member 1 ppm or less.

[0016] It is because having specified the metal impurity content as 1 ppm or less does not have enough purification of graphite material when exceeding 1 ppm, and the silicon single crystal of the high quality (high grade) demanded is not obtained with the pull-up equipment using the graphite product which consists of such a member. Few contents of a metal impurity are so good that there are.

[0017] Invention of the above (2) is the manufacture approach of the high-purity-graphite member of (1), and is the approach of heat-treating at a 1500-2500-degree C elevated temperature under reduced pressure as a high purifying method (purification processing).

[0018] Metal impurities in the graphite member made harmful to a semi-conductor by this processing, such as Fe, nickel, Cr, Cu, and Na, evaporate, and are removed from a purification processing furnace by the vacuum pump etc.

[0019] The evaporation of an impurity is decided by purification processing temperature and the degree of vacuum. For example, the relation of the full volatilization temperature of Fe which is a typical metal impurity, and a degree of vacuum is shown in a table 1. Since low temperature takes long duration theoretically in processing of a large-sized product in which industrial production of the degree of vacuum is carried out although Fe volatilizes thoroughly by heat treatment of 1595 degrees C or more by 10^{-6} Torr at 979 degrees C or more and 10^{-1} Torr etc., it is realistic to perform high temperature processing.

[0020]

[A table 1]

表 1

真空度	760Torr	10Torr	10^{-3} Torr	10^{-6} Torr
Feの完全揮発温度	2926℃	2120℃	1282℃	979℃

[0021] Although purification processing temperature changes with the classes and degree of vacuums of a metal impurity which it is going to remove, it is possible to carry out evaporation clearance of the degree of vacuum also about which the above-mentioned metal impurity, even if it is a large-scale industrial product by [suitable in 10^{-1} - 10^{-6} Torr, then a 1500-2500-degree C temperature region] carrying out time amount processing, and a metal impurity content can obtain a high grade article 1 ppm or less.

[0022] When processing temperature is less than 1500 degrees C, although evaporation of an impurity metallic element advances, it requires time amount, and in a large-sized article like the graphite member for a silicon pull-up, even if it processes for a long time, its homogeneous high grade product is difficult to get, and it is inferior also in productivity. Moreover, if it processes at the temperature exceeding 2500 degrees C, since change will arise in the degree of graphitization of a product and product properties (reinforcement etc.) will deteriorate, it is not desirable. In addition, what is necessary is just to usually make the processing time into 1 hour - 5 hours that what is necessary is just to set suitably according to the magnitude of a processing object.

[0023] By less than 10 to 1 Torr, the degree of vacuum at the time of purification processing (If it puts in another way and the pressure of a residual gas is higher than 10-1Torr), When aimed at a large-sized article, a homogeneous high grade article is difficult to get, and if it is going to obtain the degree of vacuum (condition that the pressure of a residual gas is lower than 10-6Torr) exceeding 10-6Torr, since long duration will be taken for an expensive facility to be needed and to reach a predetermined degree of vacuum, productivity also falls.

[0024] It can carry out easily [face the above-mentioned this invention approach carrying out, and a special facility is not required, and / since what is necessary is just to use a vacuum facility of a heating means (method) usually used to use carbon-resistance generation of heat, RF generation of heat, etc., a rotary pump, a diffusion pump, etc.], without being accompanied by cost rise.

[0025]

[Example] Below, the example and the example of a comparison concerning this invention are explained concretely.

[0026] After producing a graphite crucible with a bore [for a [example 1] silicon pull-up] of 18 inches through each process of shaping, baking, and graphitization, purification processing was carried out based on this invention approach. Purification processing conditions were set to 2500 degree-Cx2hr and 10-1Torr.

[0027] The core of the obtained graphite member (graphite crucible) is sampled, and the result which carried out purity assessment by neutron activation analysis is shown in a table 2. In less than (Tr expressed in the table) 0.1 ppm, each analyzed metallic element was a high grade very much.

[0028] Except [all] having set [example 2] purification processing conditions to 2000 degree-Cx2hr and 10-1Torr, the graphite member (graphite crucible) was produced according to the same process as an example 1, and purity assessment was performed by the same approach.

[0029] A result is shown in a table 2. It was a high grade very much like the case of an example 1 also in this case.

[0030] Except [all] having set [example 3] purification processing conditions to 2000 degree-Cx2hr and 10-5Torr, the graphite member (graphite crucible) was produced according to the same process as an example 1, and purity assessment was performed by the same approach.

[0031] Although the result was similarly shown in a table 2, it was a high grade very much like the case of examples 1 and 2.

[0032] Except [all] having set [example 4] purification processing conditions to 1500 degree-Cx2hr and 10-6Torr, the graphite member (graphite crucible) was produced according to the same process as an example 1, and purity assessment was performed by the same approach.

[0033] The result was a high grade very much like the case of other examples as shown in a table 2.

[0034] Except [all] having set the [example 1 of comparison] purification processing conditions to 1400 degree-Cx2hr and 10-1Torr, the graphite member (graphite crucible) was produced according to the same process as an example 1, and purity assessment was performed by the same approach.

[0035] Although the result was shown in a table 2, since processing temperature had separated from the conditions specified by this invention approach, purification was inadequate, and the metal impurity content was over 1 ppm greatly.

[0036] Except [all] the conventional elevated-temperature halogen gassing (it heating at 2000 degrees C among a chlorine gas ambient atmosphere for 3 hours) having performed the [example 2 of comparison] purification, the graphite member (graphite crucible) was produced in the same process as an example 1, and purity assessment was performed by the same approach.

[0037] The result was as having been shown in a table 2, and there were many contents of a metal impurity and purification's were inadequate.

[0038]

[A table 2]

表 2

	純化処理 条 件	金属不純物含有量 (単位ppm)						
		Fe	Ni	Cr	Cu	Na	K	Ca
実施例 1	2500℃ 10 ⁻¹ Torr	Tr	Tr	Tr	Tr	Tr	Tr	Tr
実施例 2	2000℃ 10 ⁻¹ Torr	Tr	Tr	Tr	Tr	Tr	Tr	Tr
実施例 3	2000℃ 10 ⁻⁵ Torr	Tr	Tr	Tr	Tr	Tr	Tr	Tr
実施例 4	1500℃ 10 ⁻⁶ Torr	Tr	Tr	Tr	Tr	Tr	Tr	Tr
比較例 1	1400℃ 10 ⁻¹ Torr	1.2	1.5	0.7	1.7	1.0	0.5	2.5
比較例 2	高温加熱 ガス処理	0.9	1.0	0.6	0.3	1.9	1.2	2.0

(注) Tr: 0.1ppm未満

[0039]

[Effect of the Invention] The graphite member concerning this invention has very few contents of a metal impurity, if this member is used, there is little contamination by the impurity, and it is possible to manufacture the silicon single crystal of high quality. The graphite member of this high grade can be manufactured without needing a special facility easily by this invention approach.

[Translation done.]

* NOTICES *

JPO and NCIPi are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] It is drawing showing typically the configuration of the general crystal manufacturing installation used by the CZ process.

[Description of Notations]

1: A graphite crucible, 2: support shaft, 3: heater, 4: quartz crucible, 5: melting liquid, 6: raising shaft, 7: seed crystal, 8: single crystal, 9 : heat insulating mould

[Translation done.]

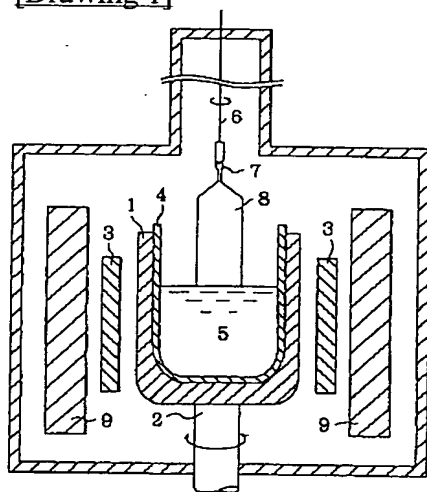
* NOTICES *

JPO and NCIPi are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

DRAWINGS

[Drawing 1]



[Translation done.]

(19) 日本国特許庁 (J P)

(12) 公開特許公報 (A)

(11) 特許出願公開番号

特開平8-337493

(43) 公開日 平成8年(1996)12月24日

(51) Int.Cl. ⁸	識別記号	庁内整理番号	F I	技術表示箇所
C 3 0 B 15/10			C 3 0 B 15/10	
// C 3 0 B 29/06	5 0 2	7202-4G	29/06	5 0 2 B
H 0 1 L 21/208			H 0 1 L 21/208	P

審査請求 未請求 請求項の数2 O L (全 4 頁)

(21) 出願番号 特願平7-148737

(22) 出願日 平成7年(1995)6月15日

(71) 出願人 000002118

住友金属工業株式会社

大阪府大阪市中央区北浜4丁目5番33号

(72) 発明者 荒堀 忠久

大阪府大阪市中央区北浜4丁目5番33号住

友金属工業株式会社内

(74) 代理人 弁理士 森 道雄 (外1名)

(54) 【発明の名称】 単結晶引上げ用高純度黒鉛部材およびその製造方法

(57) 【要約】

【構成】(1) 金属不純物 (Fe、Ni、Cr、Cu、Na等) の含有量が1ppm以下である高純度黒鉛部材 (ルツボ、ヒーター等の黒鉛製品)。

(2) 成形、焼成および黒鉛化後の黒鉛部材を 10^{-1} ～ 10^{-6} Torrの減圧下で1500～2500℃の温度域で加熱処理する。

【効果】この部材を用いれば、不純物による汚染が少なく、高品質のシリコン単結晶を製造することが可能である。この高純度の黒鉛部材は、本発明方法により容易に、かつ特別の設備を必要とせずに製造することができる。

【特許請求の範囲】

【請求項1】金属不純物含有量が1ppm以下であることを特徴とする単結晶引上げ用高純度黒鉛部材。

【請求項2】被純化材を $10^{-1} \sim 10^{-6}$ Torrの減圧下で1500～2500℃の温度域で加熱処理することを特徴とする単結晶引上げ用高純度黒鉛部材の製造方法。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明は、シリコン等の半導体物質の単結晶を製造するときに用いられる高純度の単結晶引上げ用黒鉛部材およびその製造方法に関する。

【0002】

【従来の技術】従来から、半導体物質、特にシリコン単結晶を作製するに際し、チョコラルスキー法（以下、CZ法と記す）と呼ばれる回転引上げ法が広く用いられている。

【0003】図1はCZ法で使用される一般的な結晶製造装置を模式的に示す図である。同図に示されるように、有底円筒状の黒鉛ルツボ1は所定速度で回転する支持軸2に支持されており、黒鉛ルツボ1の外側にはヒーター3および保温筒9が同心円状に配設されている。黒鉛ルツボ1の内側には、同じく有底円筒状の石英ルツボ4が設置され、石英ルツボ4の内側にはヒーター3で加熱して熔融させたシリコン原料の熔融液5が充填されている。さらに、石英ルツボ4の中心軸上には、支持軸2と同一軸心で、支持軸2と同方向または逆方向に所定の速度で回転するワイヤ等の引き上げ軸6が配設されている。そして、引き上げ軸6の先端に取り付けられた種結晶7を熔融液5の表面に接触させ、引き上げ軸6を引き上げていくことにより、熔融液5が凝固して形成される単結晶8を成長させる。

【0004】近年、LSIの高集積化に伴い、シリコンの高品質化に対する要請が強く、これに伴い、黒鉛部材の純度についても一層の高純度化が要求されている。これは、CZ法によりシリコン単結晶を製造する際、引上げ炉中の炭素材料も高温にさらされ、炭素材料中の不純物が蒸発して炉内にとどまり、シリコンの品質（純度）に大きな影響を及ぼすからである。

【0005】

【発明が解決しようとする課題】シリコン引上げ用部材としての黒鉛材は、一般には成形、焼成、黒鉛化、および純化の各工程を経て製造されている。これらの製造工程を経る間に黒鉛材中に混入した不純物は、最後の純化工程で除去されるが、この純化の方法としては、黒鉛化の後に高温ハロゲンガス処理を行うのが一般的であった。

【0006】しかしながら、上記従来の方法により得られる黒鉛材中には5～20ppmの不純物が含まれ、高品質シリコン製造用の黒鉛材としては純度が不十分であ

る。

【0007】この従来の方法は、純化効果に限界があることに加え、純化処理に強酸やハロゲン含有ガスなどの腐食性物質を用いるので特別な反応装置や廃棄物処理装置を必要とし、生産性の低下およびコストアップの要因となっていた。

【0008】黒鉛材の純度を高める方法等については、例えば、減圧下でハロゲンガスを供給しつつ加熱処理する方法、ならびにこの方法により得られた黒鉛材を各部に用いた単結晶引上げ装置が提案されている（特開昭63-79759号公報、特公平6-2637号公報および特公平7-29762号公報）。しかし、ここに開示された方法は、設備および工程が複雑で、前記のコストアップ等の課題が解決されるには至っていない。

【0009】本発明は、従来の高温ハロゲンガス処理では高純度化が不十分であることから、純化効果が優れ、金属不純物含有量が1ppm以下の高純度の黒鉛材を製造することができ、設備的にも簡易で安価な方法を提供することを課題としてなされたものである。

【0010】

【課題を解決するための手段】本発明の要旨は、下記（1）の高純度黒鉛部材および（2）のその製造方法にある。

【0011】（1）金属不純物含有量が1ppm以下であることを特徴とする単結晶引上げ用高純度黒鉛部材。

【0012】（2）被純化材を $10^{-1} \sim 10^{-6}$ Torrの減圧下で1500～2500℃の温度域で加熱処理することを特徴とする単結晶引上げ用高純度黒鉛部材の製造方法。

【0013】前記の黒鉛部材とは、図1に示したCZ法で使用される引上げ装置において、ルツボ、ヒーター、保温断熱等に用いられている黒鉛製品をいう。

【0014】また、金属不純物含有量とは、Fe、Ni、Cr、Cu、Na、K、Ca、その他不純物として含まれる全ての金属元素の合計含有量を意味する。

【0015】

【作用】本発明（前記（1）の発明）の高純度黒鉛部材は、金属不純物含有量が1ppm以下の黒鉛部材である。

【0016】金属不純物含有量を1ppm以下と規定したのは、1ppmを超える場合は黒鉛材の純化が十分ではなく、このような部材からなる黒鉛製品を用いた引上げ装置では、要求される高品質（高純度）のシリコン単結晶が得られないからである。金属不純物の含有量は少ないほどよい。

【0017】前記（2）の発明は（1）の高純度黒鉛部材の製造方法で、高純化法として減圧下で1500～2500℃の高温で加熱処理（純化処理）をする方法である。

【0018】この処理により、半導体に有害とされてい

る黒鉛部材中のFe、Ni、Cr、Cu、Na等の金属不純物は、蒸発し、真空ポンプ等によって純化处理炉から除去される。

【0019】不純物の蒸発量は純化处理温度と真空度によって決まる。例えば、代表的な金属不純物であるFeの完全揮発温度と真空度の関連を表1に示す。原理的には、真空度が 10^{-6} Torrで979℃以上、 10^{-1} Torr

表 1

真空度	760Torr	10Torr	10^{-3} Torr	10^{-6} Torr
Feの完全揮発温度	2926℃	2120℃	1282℃	979℃

【0021】純化处理温度は除去しようとする金属不純物の種類および真空度によって異なるが、真空度を 10^{-1} ～ 10^{-6} Torrとすれば、1500～2500℃の温度域で適切な時間処理することにより、大型工業製品であっても、上記のいずれの金属不純物についても蒸発除去することが可能で、金属不純物含有量が1ppm以下の高純度品を得ることができる。

【0022】処理温度が1500℃未満の場合は、不純物金属元素の蒸発は進行するが時間がかかり、シリコン引上げ用黒鉛部材のような大型品では長時間処理しても、均質な高純度製品が得難く、生産性も劣る。また、2500℃を超える温度で処理すると、製品の黒鉛化度に変化が生じ、製品特性（強度等）が劣化するので好ましくない。なお、処理時間は、処理対象物の大きさに応じて適宜定めればよく、通常は、1時間～5時間とすればよい。

【0023】純化处理時の真空度は、 10^{-1} Torr未満では（換言すれば、残留気体の圧力が 10^{-1} Torrよりも高いと）、大型品を対象とする場合、均質な高純度品が得難く、 10^{-6} Torrを超える真空度（残留気体の圧力が 10^{-6} Torrよりも低い状態）を得ようとする、高価な設備が必要となり、かつ所定の真空度に到達するのに長時間を要するので生産性も低下する。

【0024】上記本発明方法は、実施するに際し特別の設備は必要ではなく、カーボン抵抗発熱、高周波発熱等を利用する、通常用いられている加熱手段（方式）と、ロータリーポンプ、拡散ポンプ等の真空設備を用いればよいので、容易に、かつコストアップを伴わずに実施することができる。

【0025】

【実施例】以下に、本発明に係る実施例および比較例を具体的に説明する。

【0026】【実施例1】シリコン引上げ用の内径18インチの黒鉛ルツボを成形、焼成、黒鉛化の各工程を経て作製した後、本発明方法に基づき純化处理を実施した。純化处理条件は、2500℃×2hr、 10^{-1} Torrとした。

【0027】得られた黒鉛部材（黒鉛ルツボ）の中心部

*orrで1595℃以上の熱処理によりFeが完全に揮発するが、工業生産される大型製品の処理等の場合、低温では長時間を要するので、高温処理を行うのが現実的である。

【0020】

【表1】

をサンプリングし、中性子放射化分析により純度評価した結果を表2に示す。分析した各金属元素とも0.1ppm未満（表ではTrで表した）で、極めて高純度であった。

【0028】【実施例2】純化处理条件を2000℃×2hr、 10^{-1} Torrとした以外はすべて実施例1と同じプロセスにより黒鉛部材（黒鉛ルツボ）を作製し、同じ方法で純度評価を行った。

【0029】結果を表2に示す。この場合も、実施例1の場合と同様に極めて高純度であった。

【0030】【実施例3】純化处理条件を2000℃×2hr、 10^{-1} Torrとした以外はすべて実施例1と同じプロセスにより黒鉛部材（黒鉛ルツボ）を作製し、同じ方法で純度評価を行った。

【0031】結果を同じく表2に示したが、実施例1および2の場合と同様に極めて高純度であった。

【0032】【実施例4】純化处理条件を1500℃×2hr、 10^{-6} Torrとした以外はすべて実施例1と同じプロセスにより黒鉛部材（黒鉛ルツボ）を作製し、同じ方法で純度評価を行った。

【0033】結果は、表2に示すとおり、他の実施例の場合と同様に極めて高純度であった。

【0034】【比較例1】純化处理条件を1400℃×2hr、 10^{-1} Torrとした以外はすべて実施例1と同じプロセスにより黒鉛部材（黒鉛ルツボ）を作製し、同じ方法で純度評価を行った。

【0035】結果を表2に示したが、処理温度が本発明方法で規定する条件から外れているため純化が不十分で、金属不純物含有量が1ppmを大きく超えていた。

【0036】【比較例2】純化を従来の高温ハロゲンガス処理（塩素ガス雰囲気中、2000℃で3時間加熱）により行った以外はすべて実施例1と同じプロセスで黒鉛部材（黒鉛ルツボ）を作製し、同じ方法で純度評価を行った。

【0037】結果は表2に示したとおりで、金属不純物の含有量が多く、純化は不十分であった。

【0038】

【表2】

表 2

	純化処理 条 件	金属不純物含有量 (単位ppm)						
		Fe	Ni	Cr	Cu	Na	K	Ca
実施例 1	2500℃ 10 ⁻¹ Torr	Tr	Tr	Tr	Tr	Tr	Tr	Tr
実施例 2	2000℃ 10 ⁻¹ Torr	Tr	Tr	Tr	Tr	Tr	Tr	Tr
実施例 3	2000℃ 10 ⁻⁵ Torr	Tr	Tr	Tr	Tr	Tr	Tr	Tr
実施例 4	1500℃ 10 ⁻⁵ Torr	Tr	Tr	Tr	Tr	Tr	Tr	Tr
比較例 1	1400℃ 10 ⁻¹ Torr	1.2	1.5	0.7	1.7	1.0	0.5	2.5
比較例 2	高温加熱 シカス処理	0.9	1.0	0.6	0.3	1.9	1.2	2.0

(注) Tr: 0.1ppm未満

【0039】

【発明の効果】本発明に係る黒鉛部材は金属不純物の含有量が極めて少なく、この部材を用いれば、不純物による汚染が少なく、高品質のシリコン単結晶を製造することが可能である。この高純度の黒鉛部材は、本発明方法により容易に、かつ特別の設備を必要とせずに製造することができる。

*【図面の簡単な説明】

20 【図1】CZ法で使用される一般的な結晶製造装置の構成を模式的に示す図である。

【符号の説明】

1: 黒鉛ルツボ、2: 支持軸、3: ヒーター、4: 石英ルツボ、5: 熔融液、6: 引き上げ軸、7: 種結晶、8: 単結晶、9: 保温筒

【図1】

